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Effect Of Air Plasma Surface Modification on Bond Strength Between Veneering Resin and PEKK (Polyetherketoneketone)

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Abstract

Background: PEKK have been recommended by their manufacturers as a framework material for implant-supported fixed prostheses because of its high biocompatibility, light weight and compatibility with veneering materials. However, achieving high bond strength veneering composite is challenging due to PEKK's inertness and low surface energy.

Purpose: to evaluate the bond strength between PEKK and veneering composite after applying three methods of surface treatment.

Material and Methods: A total of 21 PEKK (Pekkton® Ivory, Cendres+Métaux, Bienne, Switzerland) specimens were prepared by using CAD/CAM system, embedded in acrylic resin, polished with (P180 up to P1200), and randomly divided into 3 groups (n=7) (1) Control (sandblasting with 110 µm Al_2O_3), (2) Air plasma, and (3) Sandblasting with 110 µm Al_2O_3 + air plasma. After surface pretreat, a qualitative assessment of surface roughness was done using scanning electron microscopy (SEM). With the aid of bonding jig, veneering composite was applied to each specimen and light polymerized. All completed specimens were placed in 37 °C water for 1 day for complete polymerization. Shear bond strength of each group was measured and fracture patterns were classified. Statistical analyses were carried out with One-way ANOVA followed by post-hoc LSD test.

Results: Sandblasting + Air Plasma group showed the highest mean bond strength value of (15.881 ± 1.210 MPa) followed by control group (15.048 ± 1.398 MPa) and Air plasma (9.692 ± 1.333 MPa).

Conclusion: air plasma treatment of PEKK surface showed less effect on bonding than sandblasting groups.

Introduction:

Polymers, being one of the most important materials in dentistry, offer great physical and mechanical characteristics, as well as being claimed to have high biocompatibility [(1)]. Polyaryletherketone (PAEK), is a group of thermoplastic high-performance polymers with high mechanical properties and high temperature stability. It consists of aromatic rings interconnected by ether and ketone functional groups [(2)]. PAEKs have been utilized in medicine for orthopedic applications as an implantation material for many years [(3, 4)]. In dentistry, PAEKs are considered a substitutional material to metals and ceramics due to its excellent properties such as shock absorbing ability, stress distribution and reasonable fracture resistance. PAEKs are used as a framework material for crown and bridge, clasps for removable partial dentures, and as a temporary implant abutment [(5, 6)].

The most well-known members of PAEK family are polyetheretherketone (PEEK) and polyetherketoneketone (PEKK). PEKK, which was recently released to the market for dental applications, has 80% better compressive strength and better long-term fatigue properties than the PEEK [(7, 8)]. PEKK can be heat- pressed or milled from blocks in CAD/CAM procedures [(8)]. Although PEKK possesses appealing mechanical characteristics and biocompatibility, its poor translucency and white/grayish color limit its application as a monolithic dental restorative material [(9)]. Therefore, further veneering with an esthetic material is necessary [(10)]. However, establishing a durable bond with veneering composite is challenging due to PEKK's inertness, low surface energy, and resistance to surface modification [(11, 12)]. By using the right surface treatment, you may obtain high bond strengths between PEKK and other dental materials [(13, 14)]. Plasma treatment is considered to be a well-accepted treatment method for improving bond strength. It offers effective and clean treatment that improve the surface properties while maintaining bulk properties of polymer surface [(15, 16)].

Material and Methods

Specimen preparations:

Twenty-one PEKK samples (Pekkton ivory, Cendres+Métaux, Biel-Bienne, Switzerland) were designed by ExoCAD GmbH with a dimension of 7 × 7 × 2 mm (length, width, and thickness respectively) and milled by DWX-51D Dental Milling Machine with speed not excessing 17000 rpm and 1mm Milling burs. All samples were embedded in cold cure acrylic resin (Spofa Dental Product, Czech Republic) with a dimension of 15 × 15 × 5 mm to facilitate testing procedure as in Fig. (1).

Surface treatments:

For surface standardization, all samples were polished using rotating silica carbide paper (Struers, Denmark) with four grits (P180, P320, P600 and P1200) for 60 seconds at 150 rpm under constant water cooling. Before surface treatment all specimens cleaned ultrasonically for 10 minutes with deionized water and left to dry in the air.

The PEKK samples were then randomly assessed into 3 groups (n = 7) according to surface pretreatment, as follows:

1- Control group: Sandblasting was performed for 10 seconds using 110 µm aluminum oxide particles (Shera Werkstoff Technologie, Germany) at a pressure of 2 bar and a distance of 10 mm from the nozzle to the specimen, as recommended by Pekkton company, then cleaned for 10 minutes in an ultrasonic cleaner with deionized water to remove remaining particles.

2- Air plasma group: PEKK samples are placed inside low-pressure non-thermal plasma (RF.Magnetron plasma.Iraq). The surface treatment parameters were: air gas apply for 10 minutes at a pressure of 0.3 mbar, frequency 40kHz and under 100 watts power.

3- Sandblasting + Air plasma group: The samples of this group were first Sandblasted with 110 µm Al_2O_3 similar to group (1), then treated with Air plasma like group (2).

Scanning Electron Microscopy (SEM):

Imediatly after each surface treatment, surface topography examined by SEM device (Inspect S50) at ×10,000 magnification.

Bonding procedure:
Before bonding, bonding jig was designed by SolidWorks premium 2014 and printed using the 3D printer (ender 3 pro,chine) with same dimension of specimens and 3 mm hole in the center to facilitate bonding steps, Fig. (2:A).
The bonding jig positioned on the sample and a thin layer adhesive primer (Visio.link, Bredent, Germany) was applied through 3 mm hole in center, and light-cured for 90 seconds using light-curing device (Labolight DUO). Subsequently, the 3mm hole filled with composite resin (GRADIA PLUS GUM Heavy Body, GC corporation, Japan) and before placing in light-curing device,the bonding jigs was gently removed and the specimen light-cured for 180 seconds, Fig.(2:B). For full polymerization, all bonded sepesmens were placed in water at 37 °C for one day.

Shear Bond Strength (SBS) test:
The SBS of specimens was measured using universal testing machine (Laryee technology co. LTD, China). The load of knife edge-shaped piston was applied with 1 mm/min crosshead speed, Fig. (3). The SBS calculated as follows: fracture load/bonding area (N/mm^2= MPa).

Fracture type Analysis:
Following deboning, the samples were examined under microscope (OXION, China) at a × 25 magnification to assess the type of fracture as follows:

- ❖ Adhesive fracture, meaning that no composite residues were left on the PEKK surface.
- ❖ Cohesive fracture, which occurs in the composite's bulk layer.
- ❖ mixed fracture, where composite residues are partially visible on the PEKK surface.

Statistical Methods:
The data of this study was analyzed by One-way ANOVA (post-hoc, LSD) at a significant P-value of ($P \leq 0.05$).

Results

SEM observations:
The characterization of the PEKK surface after different surface modifications was demonstrated by SEM at 10.000× magnification.
1. Control group
Sandblast treated PEKK specimens show irregular surface topography with flaws and streaks distributed along the surface, Fig. (4:A)
2. Air plasma group
Following air plasma treatment, plain and homogeneous surface topography was seen compared to the control group, Fig. (4:B)
3. Sandblasting + air plasma group
The surface topography was similar to the control group, the structure remained unchanged, fig (4:C).

SBS test:
ANOVA test and Descriptive statistics for studied groups were measured in Table (1). Sandblasting + Air Plasma group showed the highest mean bond strength value of (15.881 ± 1.210 MPa) followed by control group (15.048 ± 1.398 MPa) and Air plasma (9.692 ± 1.333 MPa) as in Fig. (5). There was a significant difference ($p<0.01$) between the groups.
Post hoc (LSD) tests were done in order to confirm the difference in shear bond strength between each experimental group, Table (2).

Fracture mode classification:
In this investigation, no cohesive fracture was observed. Air plasma group showed completely adhesive fracture while control and sandblasting + air plasma groups showed mixed and adhesive fracture, Fig. (6).

Discussion

PEKK has excellent chemical and mechanical characteristics making it ideal for dental applications, which in recent years is expanding in the use of non-metallic materials [14]. Despite PEKK's attractive properties, the interfacial binding strength to composite veneer remains a major issue, affecting long-term success of dental treatment [7]. In this study, various surface treatments are proposed to enhance the binding strength between PEKK and veneering composite.

Sandblasting increases surface roughness, removes organic contaminants, and generates an active, fresh surface. It also promotes micromechanical interlocking with the bonding agent, leading to increased bonding capacity (12). According to recent studies conducted by Pekkton®ivory manufacturer, it suggests applying MMA-based primer after sandblasting the surface using 110 µm alumina particles. Previous studies have shown that Visio.link, a primer containing MMA monomers, can achieve high bond strength between PEKK and resin composites (9, 13, 14). Moreover, the combination of sandblasting treatment with Visio.link primer was the best combination to increase PEKK adherence to composite resin (14, 17). Therefore, our study set the surface treatment parameters of the control group in line with PEKK's manufacturer instructions.

Several researches have been conducted to evaluate the efficacy of plasma surface treatment on PEKK polymer (14, 18, 19).

A study carried out by Younis et al. (2020) revealed that following air plasma treatment, substantial numbers of oxygen- and nitrogen-containing functional groups have been detected at the PEKK surface (19). That was because the reactive oxygen and nitrogen species were simultaneously present in air plasma. These functional groups responsible for the increase in surface polarity with high surface energy and hydrophilicity of polymer surface. However, our study shows a mean SBS value of (9.692 ± 1.33 MPa) which is comparable to the results by Younis et al. (2020) who reported a mean SBS value of (7.09 ± 1.99 MPa) after air plasma treatment. The reported SBS value in that study showed a significant increase in SBS in contrast to our study which showed statistically decrease in SBS. This difference was explained by the fact that they compared the results with untreated PEKK surface while the present study compared the results with sandblasted PEKK surface. Regarding etching effect, air plasma demonstrated a plain surface with no impact on surface topography as supported by SEM image, Fig. (4:B).

Moreover, studies concerning the use of air plasma in combination with sandblasting for the surface treatment of PEKK are very limited. As a result, there haven't been enough studies to compare our results to their results. The only study is by Labriaga et al. (2018), who evaluated the SBS between PEKK and resin cement and found that air plasma combined with sandblasting achieved significantly higher SBS value (17.7 ± 1.1 MPa) among the other tested groups. The current result does not confirm their results since no statistically significant improvement in SBS was achieved after using air plasma in combination with sandblasting when compared to the control group (15.881 vs. 15.048 MPa, respectively). These differences may be attributed to the different plasma treatment parameters used and they evaluated resin cement bonding to PEKK whereas we evaluated veneering composite. Also, SEM image, Fig. (4:C) showed that air plasma has no impact on sandblasted PEKK surface.

According to the guidelines of ISO 10477, the minimum acceptable SBS value is 5 MPa between resin-based materials and substrate. However, it has been reported that the minimum SBS value should be 10 to 12 MPa to provide a clinically acceptable bond strength under oral conditions (20). In this study, all the studied groups displayed SBS value greater than 5 MPa, but the clinically acceptable SBS value was not reached in Air plasma group. Also, the adhesion strength between the veneering resin and PEKK substrate is indicated by the amount of failures between them (7). Adhesive and mixed failures was the predominant types of failure with no cohesive failure. However, when the bond strength is not sufficiently high, adhesive failure can take place which is seen in air plasma group. On the other hand, mixed failure was due to the irregular distribution of forces at the interface which is seen in control group and sandblasting + air plasma group. The failure patterns support the results of this study, as the bond strength increases, the failure pattern tended to shift from adhesive to mixed failure which agree with Ates et al. (2018).

Conclusions

Within the limitations of this study, air plasma treatment of PEKK surface showed acceptable SBS value according to ISO 10477 But it has less effect compared to the sandblast group. Farther study are needed to determine best proceeding gas to achieve batter bonding strength.

Acknowledgement

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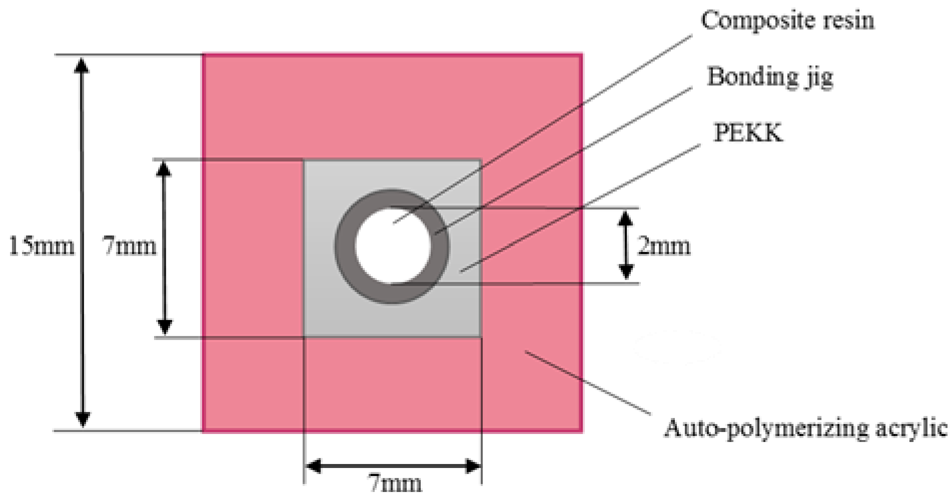

Fig. (1): Schematic drawing of specimen preparation.

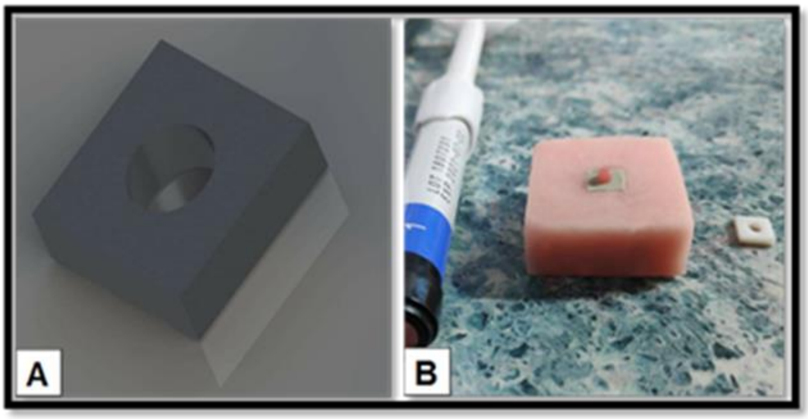

Fig. (2): A: bonding jig designing, B: Finished specimen

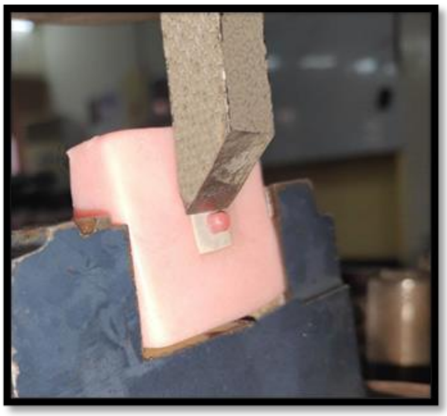

Fig. (3): SBS testing

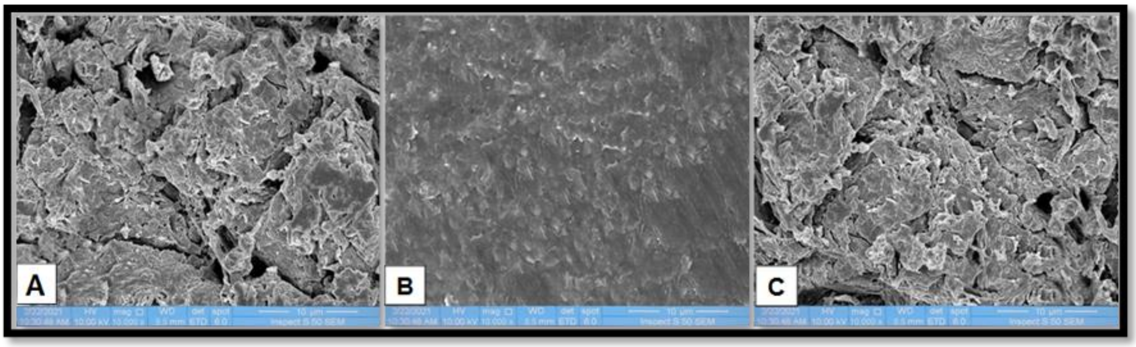

Fig. (4): SEM image of the PEKK surface after surface treatments, A: Sandblasting, B: Air plasma, C: Sandblasting + air plasma

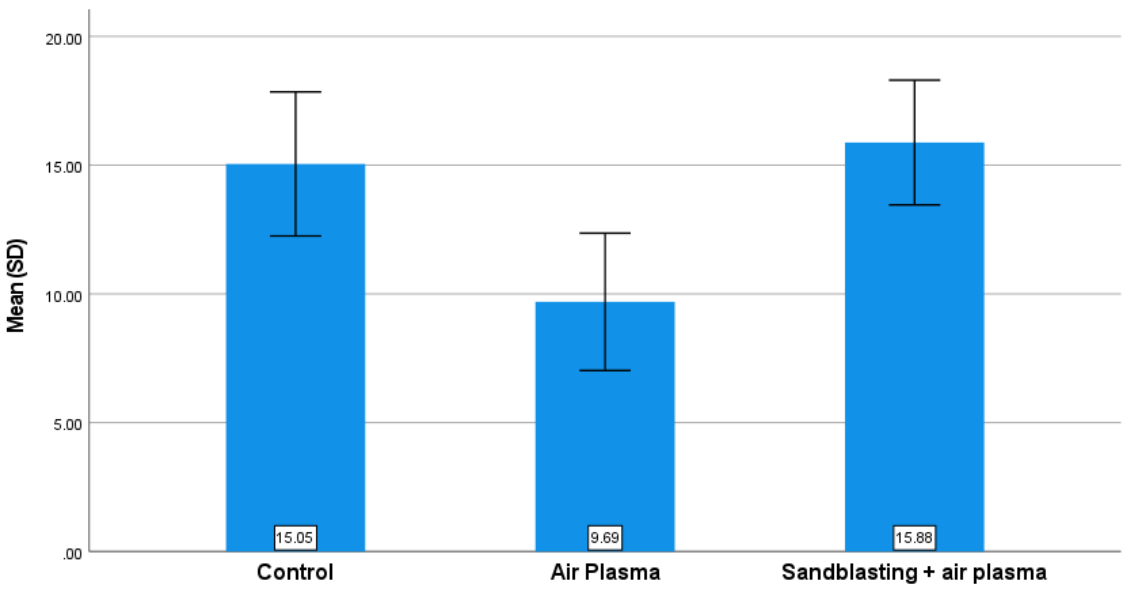

Fig. (5): Bar chart represents the mean value of SBS of the studied groups

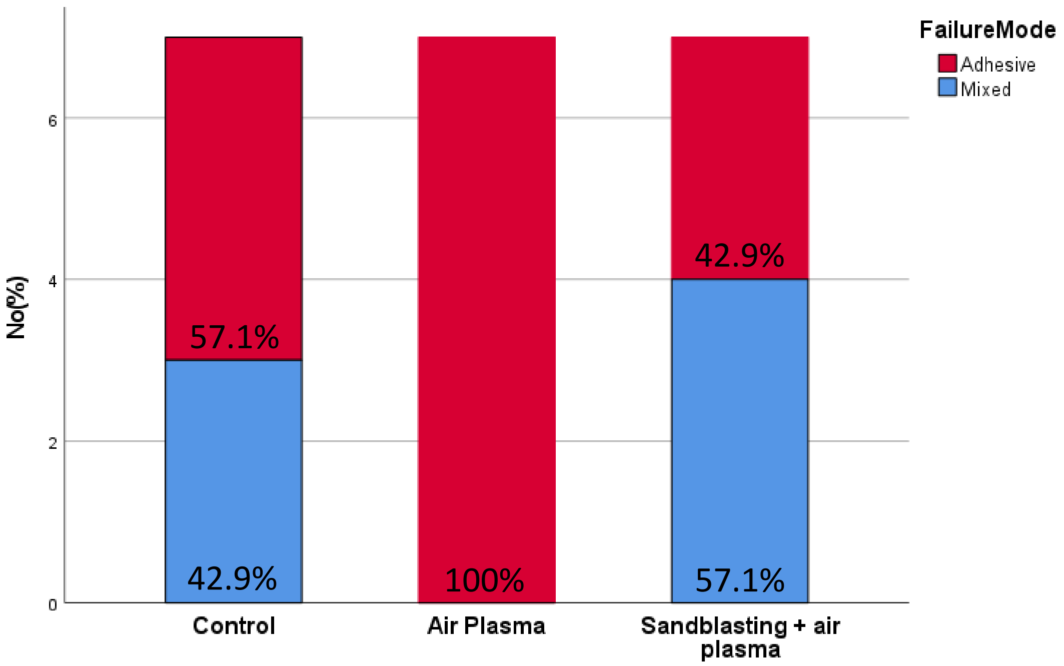

Fig. (6): Fracture modes after SBS test.

Table (1): Descriptive statistics and ANOVA test.

Studied groups	No.	Mean	SD	SE	Min.	Max.	ANOVA test
Control	7	15.048	1.398	.528	13.43	17.68	.001 significant (P<0.01)
Air Plasma	7	9.692	1.333	.503	8.48	12.02	
Sandblasting + air plasma	7	15.881	1.210	.457	14.14	17.68	
Total	21						

Table (2): Post hoc (LSD) tests for the studied groups.

section	(I) Groups	(J) Groups	Mean Difference (I-J)	P-value	Sig.
1	Control	Air Plasma	5.355*	.000	S
		Sandblasting + air plasma	-.8328	.255	NS
2	Air Plasma	Control	-5.355*	.000	S
		Sandblasting + air plasma	-6.188*	.000	S
3	Sandblasting + air plasma	Control	.8328	.255	NS
		Air Plasma	6.188*	.000	S

References

1. Alqurashi H, Khurshid Z, Yaqin SAU, Habib SR, Rokaya D, Zafar MSJJoAR. Polyetherketoneketone (PEKK): An emerging biomaterial for oral implants and dental prostheses. 2020.
2. Elmougy A, Schiemann AM, Wood D, Pollington S, Martin NJDm. Characterisation of machinable structural polymers in restorative dentistry. 2018;34(10):1509-17.
3. Fuhrmann G, Steiner M, Freitag-Wolf S, Kern MJDM. Resin bonding to three types of polyaryletherketones (PAEKs)—durability and influence of surface conditioning. 2014;30(3):357-63.
4. Ates SM, Caglar I, Yesil Duymus ZJJoAS, Technology. The effect of different surface pretreatments on the bond strength of veneering resin to polyetheretherketone. 2018;32(20):2220-31.
5. Prechtel A, Reymus M, Edelhoff D, Hickel R, Stawarczyk BJDM. Comparison of various 3D printed and milled PAEK materials: Effect of printing direction and artificial aging on Martens parameters. 2020;36(2):197-209.
6. Lee K-S, Shin J-H, Kim J-E, Kim J-H, Lee W-C, Shin S-W, et al. Biomechanical evaluation of a tooth restored with high performance polymer PEKK post-core system: a 3D finite element analysis. 2017;2017.
7. Fokas G, Guo CY, Tsoi JKJJotmbobm. The effects of surface treatments on tensile bond strength of polyether-ketone-ketone (PEKK) to veneering resin. 2019;93:1-8.
8. Dawson JH, Hyde B, Hurst M, Harris BT, Lin W-SJTJopd. Polyetherketoneketone (PEKK), a framework material for complete fixed and removable dental prostheses: A clinical report. 2018;119(6):867-72.
9. Lee K-S, Shin M-S, Lee J-Y, Ryu J-J, Shin S-WJTjoap. Shear bond strength of composite resin to high performance polymer PEKK according to surface treatments and bonding materials. 2017;9(5):350.
10. Sakihara M, Taira Y, Sawase TJO. Effects of sulfuric and vinyl sulfonic acid etchants on bond strength of resin composite to polyetherketoneketone. 2019;107(2):158-64.
11. Jun JH, Oh KC, Moon HS. Maxillary Implant-supported Hybrid Prosthesis Fabricated using a Polyetherketoneketone Framework: A Case Report. 2020.
12. Gama LT, Duque TM, Özcan M, Philippi AG, Mezzomo LAM, Gonçalves TMSVJDM. Adhesion to high-performance polymers applied in dentistry: A systematic review. 2020;36(4):e93-e108.
13. Hong MG, Shin S-YJJoDR, Science A. Comparative study of surface modification on bond strength of polyetherketoneketone adhesively bonded to resins for temporary restoration. 2020;36(1):1-11.
14. Stawarczyk B, Silla M, Roos M, Eichberger M, Lümkemann NJJAD. Bonding behaviour of polyetherketoneketone to methylmethacrylate-and dimethacrylate-based polymers. 2017;19:331-8.
15. Liu C, Bai J, Wang Y, Chen L, Wang D, Ni S, et al. The effects of three cold plasma treatments on the osteogenic activity and antibacterial property of PEEK. 2020.
16. Chen M, Zhang Y, Driver MS, Caruso AN, Yu Q, Wang YJDM. Surface modification of several dental substrates by non-thermal, atmospheric plasma brush. 2013;29(8):871-80.
17. Gouveia DdNM, Razzoog ME, Sierraalta M, Alfaro MFJTJoPD. Effect of surface treatment and manufacturing process on the shear bond strength of veneering composite resin to polyetherketoneketone (PEKK) and polyetheretherketone (PEEK). 2021.
18. Labriaga W, Song S-Y, Park J-H, Ryu J-J, Lee J-Y, Shin S-WJTjoap. Effect of non-thermal plasma on the shear bond strength of resin cements to Polyetherketoneketone (PEKK). 2018;10(6):408.
19. Younis M, Unkovskiy A, Drexler T, Qian J, Wan G, Spintzyk SJJotMBoBM. The impact of non-thermal plasma on the adhesion of polyetherketoneketone (PEKK) to a veneering composite system. 2020;112:104065.
20. 10477 I. Dentistry–Polymer-based crown and bridge materials. International Standards Organization (ISO) Geneva, Switzerland; 2004.